A GC-MS database of target compound chromatograms for the identification of arson accelerants

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Science & Justice 1995; 35(1): 19–30
Received 18 June 1993; accepted 10 January 1994

Conventional gas chromatographic analysis of highly contaminated fire debris samples often gives ambiguous results with respect to the presence or otherwise of an accelerant. This is typically the result of interfering compounds produced by the pyrolysis of the matrix material itself. A gas chromatography/mass spectrometry (GC-MS) technique is described which largely overcomes this problem of background interference. It involved a target compound procedure where the information on forty selected components of petroleum products was automatically retrieved from the total ion chromatogram of the sample by the GC-MS software. This information was used to construct a "target compound chromatogram" or TCC. A database of accelerant TCCs and a user-generated search program have proved to be valuable aids for the interpretation of chromatographic profiles.

L'analyse par chromatographie en phase gazeuse conventionnelle de débris d'incendie hautement contaminé, donne souvent des résultats ambigus en ce qui concerne la présence ou non d'un accélérant. Typiquement, c'est le résultat de composés d'interférence produits par la pyrolyse du matériel de la matrice support. Une technique de chromatographie en phase gazeuse liée à la spectrométrie de masse (GC-MS) évite grandement ce problème d'interférence de fonds. Celle-ci consiste en la recherche de composés cibles. L'information concernant 40 composés dérivés du pétrole sélectionnés sont automatiquement cherchés dans un chromatogramme d'ions total d'un échantillon par le programme informatique du GC-MS. Cette information a été utilisée pour construire un chromatogramme de composés cibles ou TCC. Une base de données des TCC d'accélérants et un programme de recherche généré par l'utilisateur a démontré que c'était une aide considérable pour l'interprétation des profils chromatographiques.

Konventionelle Gaschromatograficanalysen von stark kontaminierten Brand-Asservaten ergeben oft unklare Resultate in bezug auf das Vorhandensein möglicher Brandbeschleuniger. Dies ist typisch der Fall, wenn Pyrolyse-Produkte des Spurenträgermaterials die Analysen stören. Mittels einer speziellen Gaschromatografie/Massenspektrometrie (GC-MS) Technik können diese Probleme gelöst werden. Sie beinhaltet ein Analysen-Vorgehen, bei welchem die Information von 40 ausgewählten Verbindungen von Erdoelprodukten mit Hilfe der GC-MS-Software automatisch auf Vorhandensein in den Chromatogrammen überprüft werden. Die Ergebnisse werden sodann benutzt, um das Zielmaterial-Chromatogramm zu erstellen. Eine Datenbank möglicher Brandbeschleuniger und ein benutzergeneriertes Suchprogramm haben sich als nützlich erwiesen für die Interpretation chromatografischer Profile.

La cromatografia de gases convencional de muestras de residuos de incendios, que presentan un alto grado de impregnación produce a menudo resultados ambiguos respecto a la presencia o ausencia de un acelerante. Esto es resultado típico de las interferencias de compuestos producidos por la pirólisis del propio material. Se describe una técnica de cromatografia de gases-espectrometría de masas (CG-EM) capaz de superar este problema de las interferencias de fondo. Consiste en un procedimiento de información de cuarenta compuestos diana correspondientes a productos derivados del petróleo que son automáticamente seleccionados por el software del CG-EM. Esta información se usa para construir un "cromatograma de compuestos diana" llamado "TCC". Se ha demostrado la utilidad del uso de una base de datos sobre "TCCs de aceleradores en combinación con un programa de búsqueda para la interpretación de perfiles cromatográficos.

Key Words: Fire investigation; Arson; Accelerants; GC-MS; Target Compound Chromatography; Fire debris.

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Introduction

Arson is a crime of increasing occurrence and economic impact. Added to the direct cost of buildings and furnishings lost to deliberately lit fires are costs such as injury, death and lost income. The criminalist is faced with the fact that a fire will tend to destroy any physical evidence left by the criminal. However, in cases where an inflammable liquid has been used, enough unburned material may remain absorbed in the fire debris to be detected and identified by certain analytical techniques. The detection of an accelerant in a fire sample can indicate criminal activity and provide important evidence regarding the intentional element of the crime.

The isolation and detection of volatile residues in fire debris is an interesting analytical problem faced by the forensic chemist. The first problem encountered is the physical isolation of very small quantities of accelerant from the sample of heterogeneous, charred debris taken from the fire scene. Static or dynamic (adsorption-elution) headspace techniques are typically used to perform this task [1]. The second problem involves the detection and identification of the accelerant in the presence of compounds resulting from the debris itself (pyrolysis products, natural resins, etc.).

Petroleum-based fuels such as petrol (gasoline), lighter fluids and kerosene tend to be widely used by arsonists. Hydrocarbon fuels such as these may be classified according to their alkane and aromatic components. Gas chromatography (GC) using an inexpensive, non-specific flame ionization detector (FID) remains the technique of choice for the routine analysis of such materials. Typically, the chromatogram recorded for an unknown fire-related sample is compared with a collection of standard chromatograms of known inflammable materials. In ideal cases, a visual comparison is sufficient to determine the presence or absence of a hydrocarbon accelerant and to classify an accelerant according to its chromatographic profile.

The interpretation of chromatographic profiles is greatly complicated by the presence of pyrolysis products resulting from the combustion of synthetic materials (carpets, fabrics, foam padding, floor tiles, etc.). Depending on the polymer type, combustion can result in the formation of volatile compounds such as toluene, xylene, styrene, amines, cyanides, isocyanates and other complex volatiles [2]. A fire-related sample may therefore produce a chromatogram containing accelerant peaks partially or totally obscured by peaks

due to the fire debris itself. A visual comparison of such chromatograms with those from standard accelerants can thus lead to serious misinterpretations. An accelerant can go undetected or be incorrectly classified; pyrolysis products may be misidentified as accelerant components.

GC-MS analysis of arson samples

The replacement of the non-specific FID with a mass spectrometer (MS) can greatly facilitate the interpretation of chromatographic profiles. A single peak in the total ion chromatogram (TIC) can be isolated and the compound identified from both its retention time and mass spectrum. Alternatively, extracted ion chromatograms can be constructed in order to obtain profiles for certain groups of compounds. In this manner, peaks can be eliminated that do not contain certain ions, thus greatly simplifying the chromatograms.

Nowicki [3, 4] recently illustrated the use of extracted ion chromatography (or "mass chromatography") for the classification of hydrocarbon accelerants isolated from fire debris samples. With his method, extracted ion chromatograms were constructed for six different compound groups: alkanes, cycloparaffins, aromatics, naphthalenes, dihydroindenes and terpenes. The resulting ion profiles greatly simplified the analysis of fire debris samples for the presence of petroleumbased accelerants. Vella [5] reported a similar technique using a GC fitted with an ion trap detector (ITD) and found that ion profiling enabled a more confident appraisal to be made of samples having significant background interference from matrix contributions. However, this technique could not eliminate peaks due to pyrolysis products or other materials in the matrix in cases where these products fell into one of the particular compound groups under study.

Another approach to the GC-MS analysis of arson samples has been proposed by Keto and Wineman [6]. Their method involved the detection and quantitation of a number of selected petroleum-related target compounds. These may be identified by GC-MS even in highly contaminated extracts of fire debris. The TIC of a particular sample was treated by a special data analysis program that looked for GC peaks at specific retention times, then checked the identity of each target compound by considering the relative abundance of 2 or 3 characteristic m/z ions. The technique could effectively separate compounds of interest from coeluting volatiles originating from the substrate itself. Two different GC analysis methods and three different

identification files were used to cover the three main classes of hydrocarbon accelerants: petrol (gasoline), medium petroleum distillates (MPD; lighter fluids, paint thinners, etc.), and heavy petroleum distillates (HPD; diesel fuel, kerosene, etc.). Semiquantitative data were obtained for the target compounds of each group and were used to construct a "target compound chromatogram" (TCC), i.e., a bar graph of base ion peak area versus retention time for each target compound identified. Keto and Wineman compared these TCCs with those generated from standard petroleum samples and showed that background patterns for pyrolysis products common to fire scenes were dissimilar to petroleum profiles and therefore posed no risk of false identification. The advantages of the technique were illustrated by the extraction of usable target compound profiles from highly contaminated accelerant samples.

The target compounds chosen by Keto and Wineman were those known to be present in the accelerants under investigation and which remained detectable when the accelerant was highly evaporated, diluted, and contaminated with high levels of coeluting substances. Components more volatile than nonane were excluded for lack of persistence at fire scenes and to avoid interference from simpler molecules that are often produced by substrate pyrolysis. Certain compounds were added to guard against false positives while others, such as naphthalene, were found to be too common in pyrolysis products to be useful. Fifteen target compounds were chosen for the identification of petrol (gasoline) samples, 15 target compounds for medium petroleum distillates, and 26 for heavy petroleum distillates (in total, 40 different compounds in three separate files). The ions chosen for identification purposes consisted of the base ion for each target compound with one or two other strong ions, added to increase selectivity.

Despite the obvious advantages of the TCC method as proposed by Keto and Wineman, a number of disadvantages may be considered. Firstly, before analyzing the fire debris sample by GC-MS, the forensic chemist must select the appropriate chromatographic method (either for petrol/MPD samples or for HPD samples) and the appropriate identification file for petrol, MPD or HPD. Results obtained by GC-FID analysis may suggest, in most cases, what method and identification file should be used. On the other hand, some chromatograms obtained from the GC-FID analysis of fire debris samples do not clearly indicate the presence or otherwise of an accelerant, let alone permit a tentative classification. Secondly, the

TCC generated by the GC-MS software for a particular sample must be visually compared with a printed database of TCCs recorded for standard accelerants. Visual comparisons, even made by experienced technicians, always contain some degree of subjective assessment.

In this report, a modified TCC technique is described which is aimed at overcoming the disadvantages indicated for the GC-MS procedure proposed by Keto and Wineman. A single GC method was developed, capable of analyzing any of the common petroleum-related accelerants. The three separate identification files were combined into one single file, containing 40 target compounds in total. TCC data generated from the analysis of pure, evaporated and partially burnt petroleum products were stored in an Excel database. An Excel macro was developed to search automatically the TCC obtained from a suspect fire sample to establish the presence and identity of an accelerant.

Experimental procedure

Sample preparation

Various hydrocarbon fuels (petrol, charcoal lighter fluid, white spirit, paint thinners, kerosene, diesel, etc.) were obtained from local suppliers. These samples were analyzed either as received, after artificial weathering, or after controlled combustion on a variety of substrates (wood, carpet, clothing, plastic, painted surfaces, etc.). The weathering was achieved by evaporation of the accelerant to either 50, 20 or 10% of the original volume.

The accelerant sample, either in the liquid form deposited on a cloth support or as the fire debris itself, was sealed in a nylon bag (Rilsan) and heated in an oven at 100 °C for 30 minutes. The nylon bag was then pierced with a hypodermic needle connected to a charcoal sampling tube (ORBO-32 small tubes containing 150 mg activated coconut charcoal, mesh size 20/40; Supelco, Bellefonte, PA). This tube was in turn connected to a vacuum pump adjusted to provide a suction rate of approximately 300 ml/min. Dynamic headspace sampling, in this manner, was conducted over 15 minutes with the sample maintained at 100 °C. The adsorbed volatiles were then eluted from the charcoal tube with 0.5 ml of carbon disulphide (Fluka Chemie AG, Buchs). The final extracts were stored at 7°C in sealed 1 ml glass vials before GC-MS analysis.

GC-MS analysis

The GC-MS system consisted of a Hewlett-Packard 5890 Series II gas chromatograph connected to a Hewlett-Packard 5971A mass selective detector (Hewlett-Packard, Avondale, PA). Data analysis was performed on a Hewlett-Packard Vectra QS/20 personal computer using MS Chemstation software, version G1034B (Hewlett-Packard, Avondale, PA). Additional processing was performed with Microsoft Excel spreadsheet software, version 2.1 (Microsoft Corporation, Redmond, WA). Operating parameters for the GC-MS analysis are listed in Table 1. Sample injections from 5 to 20 μ l were used depending on the accelerant concentration in the final CS2 extract.

TABLE 1 GC-MS operating parameters

Column	DB-5, nonpolar capillary, 0·25 mm i.d. × 30 m, 1 μm thick bonded phase (J&W Scientific, Folsom, CA) helium, 55 kPa column head pressure, 0·9 ml/min, 30 cm/sec linear velocity				
Carrier gas					
Injector	split/splitless in splitless mode, 250 °C				
Transfer line	280 °C				
GC program					
initial temp.	70 °C (zero hold)				
final ramp rate	2 °C/min				
first intermediate temp.	130 °C (zero hold)				
second ramp rate	5 °C/min				
second intermediate temp.	260 °C				
second intermediate hold	4 min				
third ramp rate	10 °C/min				
final temp.	280 °C				
final hold	13 min				
total run time	75 min				
Mass spectrometer					
MS on	5 min (solvent delay)				
MS off	60 min				
scan range	50–200 amu				
scan cycle	1.2 sec (16 A/D Samples)				
threshold	500 counts				
ionization	electron impact				

Raw GC-MS data were quantitated using a target compound identification file as shown in Table 2. The indicated ion intensities (relative abundances) reflect the actual performance of the instrument during the GC-MS run with an accepted tolerance of $\pm 20\%$ for each ion. The retention time for each compound is an absolute value as measured on our system with an accepted tolerance of ± 0.3 min. These values must be

recalibrated with a test mixture if significant changes to the analysis conditions (flow rate, column, temperature, etc.) are made.

The resulting peak area data were transferred to the spreadsheet program and used to construct the target compound chromatogram. TCCs of the various petroleum products (fresh, evaporated, and burnt on various supports) were collected and the values stored in a spreadsheet database after normalization. For each sample, the relative amounts for each of the 40 target compounds were recalculated so that the total for all the 40 compounds combined equalled 10 000 (arbitrary units). This normalization is required to compensate for differences in sample concentration and injection volume. A user-generated macro was employed to search unknown accelerants against the standard samples contained in the database. The program calculates a "match" value by adding up the absolute differences between the normalized quantities of each target compound. A zero value therefore corresponds to a perfect match.

Results and discussion

Accelerant profiles

A total ion chromatogram (TIC) and the corresponding target compound chromatogram (TCC) for a typical accelerant (petrol) are shown in Figure 1. The numbers on the TCC denote target compounds as listed in Table 2.

It is important to note the difference between the chromatographic profiles obtained after dynamic headspace sampling, as conducted in this study (Figure 2a), and those obtained by direct injection of the liquid petroleum products. Figure 2b illustrates the results obtained from the GC-MS analysis of a diesel sample, injected after prior dilution with pentane. The TIC and TCC profiles are significantly different from those obtained by headspace sampling but in good agreement with those obtained by Keto and Wineman [6]. These authors analyzed fresh and weathered accelerants by direct injection into the chromatograph (after dilution to approximately 1% with carbon disulphide). The heavier hydrocarbon components (particularly the last three target compounds, nonadecane, eicosane and heneicosane) were easily detected by direct injection but absent when headspace sampling was used. Their results therefore indicate the importance of constructing a reference library of TCCs using the same conditions as those employed for routine casework samples. In the present paper, this involved sampling by a dynamic

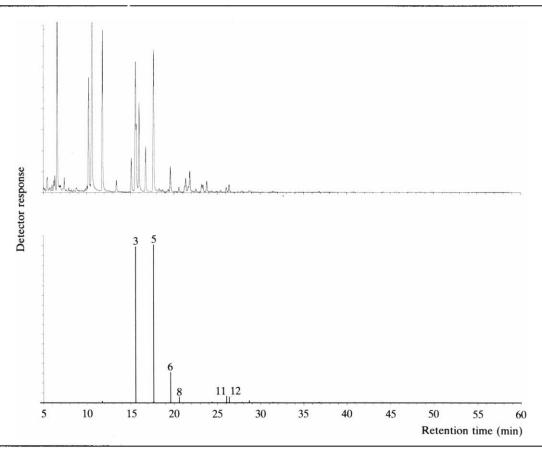


FIGURE 1 Total ion chromatogram (above) and target compound chromatogram (below) for a fresh petrol sample. The numbers refer to target compounds listed in Table 2.

headspace procedure using activated charcoal as an adsorbant and carbon disulphide as an extracting solvent. Standard sample preparation and analysis conditions were maintained throughout this work in order to limit variations in chromatographic profiles.

The effects of artificial weathering are illustrated for a lighter fluid sample analyzed as received (Figure 3), after 50% evaporation (Figure 4a), and after evaporation to 10% of its original volume (Figure 4b). As an accelerant evaporates, either during a fire or by artificial weathering in the laboratory, the more volatile components are the first to be lost, resulting in the enhancement of later eluting GC peaks. This phenomenon is evident in the illustrated chromatograms and associated TCCs. As stressed by Keto and Wineman [6], it is essential to have a reference library of TCCs for accelerants in various stages of evaporation.

Reproducibility and sensitivity

Repeated GC-MS analysis of similarly prepared petrol, white spirit and diesel samples indicated a high

level of reproducibility for the TCC profiles obtained. Using the database search macro, mean match values of less than 1000 were obtained for each accelerant type. It was determined that a match value of less than 2000 constituted good evidence for the identification of a given accelerant sample.

Samples of accelerant down to $1\cdot 0\,\mu l$, placed on a cloth support and sealed in a nylon bag, could be readily detected and identified by the described adsorption-elution/GC-MS technique. Below this level, background noise tended to mask the accelerant chromatogram. For similarly prepared samples, this detection limit corresponded to approximately ten times the minimum volume required for conventional GC-FID analysis.

Accelerants analysis after combustion

Tests involving the combustion of several different petroleum products on a variety of substrates (wood, carpet, clothing, plastic, synthetic foams, painted surfaces, etc.) illustrated the ability of the GC-MS to extract usable target compound profiles from highly

TABLE 2 GC-MS target compound identification file

No.	Target compound	Retention time (min.)	m/z of ion	relative abundance	No.	Target compound	Retention time (min.)	m/z of ion	relative abundance
1	nonane	11.7	57	100	21	tridecane	36.9	57	100
•	nonane	11 /	85	60				71	60
			71	30				85	40
2	propylcyclohexane	13.9	83	100	22	2-methylnaphthalene	37.2	142	100
_	propyroyerenemano	,	55	90				141	90
3	1,3,5-trimethylbenzene	15.6	105	100	23	1-methylnaphthalene	38-1	142	100
•	-,e,e		120	30				141	90
4	decane	17.6	57	100	24	n-heptylcyclohexane	38.9	83	100
			71	60				82	60
			85	40	25	tetradecane	40-8	57	100
5	1,2,4-trimethylbenzene	17.7	105	100				71	60
	•		120	40				85	40
6	1,2,3-trimethylbenzene	19.5	105	100	26	ethylnaphthalenes (mixed)	41.3	141	100
	•		120	40	27	1,3-dimethylnaphthalene	41.7	156	100
7	n-butylcyclohexane	20.1	83	100				141	70
	3 3		82	70	28	2,3-dimethylnaphthalene	42.3	156	100
8	indane	20.5	117	100				141	70
			118	70	29	n-octylcyclohexane	42.7	83	100
			115	50				82	50
9	trans-decalin	22.2	138	100	30	pentadecane	44.1	57	100
			96	80				71	80
			81	50				85	40
10	undecane	24.3	57	100	31	2,3,5-trimethylnaphthalene	45.8	170	100
			71	50	32	n-nonylcyclohexane	46.0	83	100
			85	40				82	80
11	1,2,4,5-tetramethylbenzene	26.1	119	100	33	hexadecane	47.0	57	100
	•		134	40				71	70
12	1,2,3,5-tetramethylbenzene	26.3	119	100				85	40
	•		134	40	34	heptadecane	49.7	57	100
13	n-pentylcyclohexane	27.3	83	100				71	70
			82	80				85	50
			55	70	35	pristane	49.9	57	100
14	5-methylindane	27.8	117	100				71	80
	•		132	40				85	30
15	4-methylindane	28.6	117	100	36	octadecane	52.1	57	100
	ř		132	40				71	80
16	C ₁₁ hydrocarbon	28.9	57	100				85	50
	,		71	60	37	phytane	52.4	57	100
17	dodecane	31.4	57	100				71	70
			71	50				85	40
			85	30	38	nonadecane	54.4	57	100
18	C ₁₂ hydrocarbon	32.3	57	100				71	70
			71	40				85	50
19	n-hexylcyclohexane	34.1	83	100	39	eicosane	56.5	57	100
			82	90				71	60
			55	70				85	40
20	4,7-dimethylindane	34.9	131	100	40	heneicosane	58-6	57	100
	•		146	30				71	70
								85	40

contaminated accelerant samples. In all cases, the TCC profile obtained and the match value calculated by the database search program left little doubt as to the presence and identity of the accelerant.

The chromatograms from the analysis of the debris resulting from the combustion of a small quantity of petrol (5 ml) on a mixture of wood and plastic materials are illustrated in Figure 5 with the associated

database search results. Despite the presence of a number of peaks due to pyrolysis of the matrix, the accelerant could be readily detected and identified as petrol. Similarly, TIC and TCC chromatograms from the combustion of a diesel sample (1 ml) on a mixture of wood, synthetic foam and assorted plastic materials, are presented in Figure 6. The database search results and the general TCC profile leave no doubt as to the identity of the accelerant used.

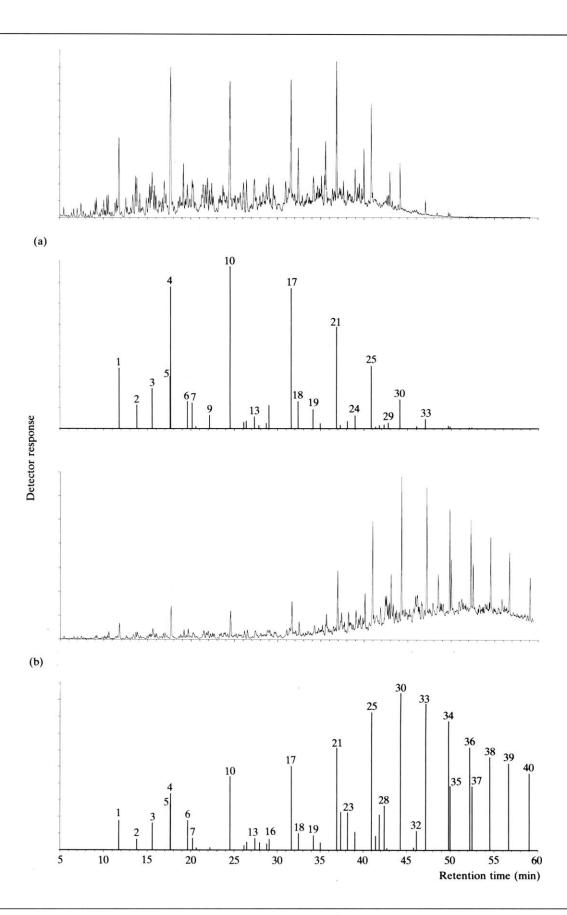


FIGURE 2 Total ion chromatogram and target compound chromatogram for (a) a fresh diesel sample and (b) a fresh diesel sample analyzed by direct injection. The numbers refer to target compounds listed in Table 2.

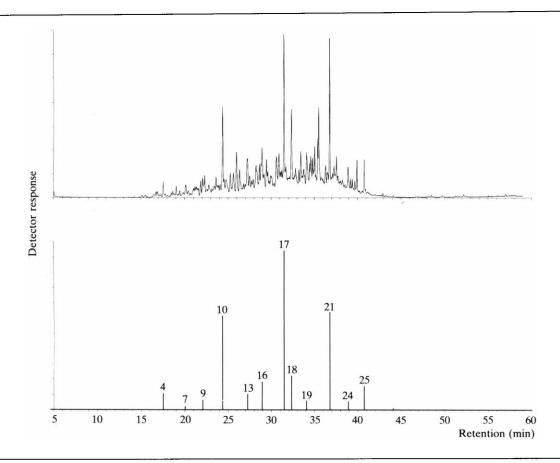


FIGURE 3 Total ion chromatogram and target compound chromatogram for a fresh, non-evaporated lighter fluid sample. The numbers refer to target compounds listed in Table 2.

Pyrolysis samples, prepared by the combustion of various materials (wood, carpet, plastic, etc.) but without added accelerant, were analyzed in order to determine potential interferences with the target compound method. The resulting chromatographic profiles showed little resemblance to the TCCs of common petroleum fractions. When searched against known accelerants, these pyrolysis samples gave very large match values and posed no risk of a false identification. This confirmed the observations reported by Keto and Wineman [6].

Casework example

The target compound method was successfully applied to casework samples in instances where the GC-FID analysis of a fire debris sample had given ambiguous results. Figure 7 illustrates such a case where the conventional analysis of a sample of partially carbonized material failed to identify unequivocally an accelerant, due to the presence of numerous matrix-related peaks. The TCC profile and the database search results indicated the presence of

weathered petrol. This was confirmed by comparison of the chromatograms with the results obtained from the analysis of an artificially weathered (90% evaporated) petrol sample (Figure 8).

Conclusions

A modified target compound GC-MS technique has been successfully used for the detection and classification of petroleum-based accelerants in fire debris samples. In addition, a database of target compound chromatograms for fresh, weathered and highly contaminated accelerants has been constructed using commercially available spreadsheet software. This database, which presently contains TCC data for over 180 samples, may be conveniently searched via a user-generated macro program. In this way, the TCC profile of a fire debris sample can be matched against those contained in the database. The results can indicate the presence and identity of residual petroleum products. This system has already proved to be of significant value in actual casework when conventional GC-FID analysis has given ambiguous

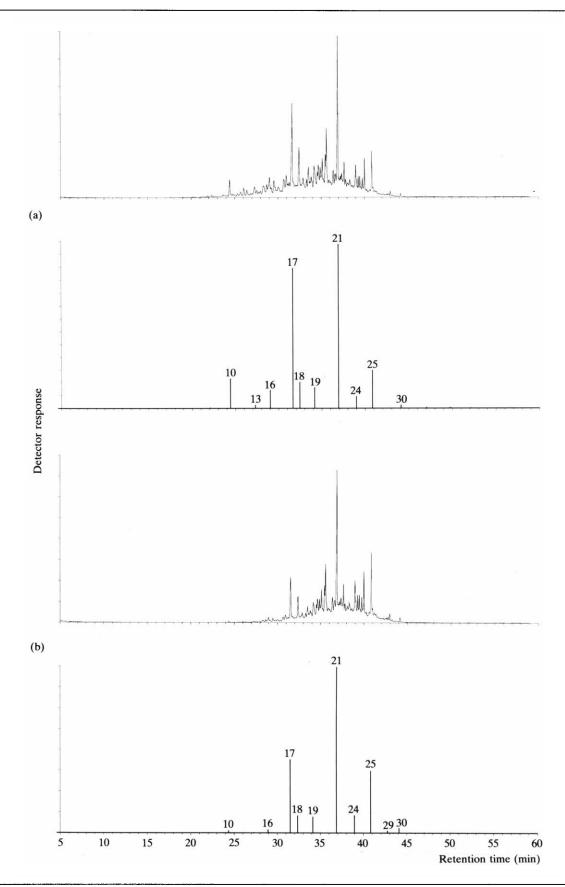


FIGURE 4 Total ion chromatogram and target compound chromatogram for (a) a 50% evaporated lighter fluid sample and (b) a 90% evaporated lighter fluid sample. The numbers refer to target compounds listed in Table 2.

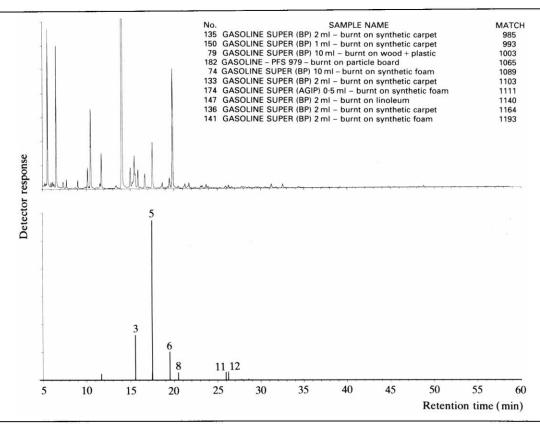


FIGURE 5 Total ion chromatogram and target compound chromatogram for a petrol sample after combustion on a mixture of wood and assorted plastic materials. Insert shows database search results. The numbers refer to target compounds listed in Table 2.

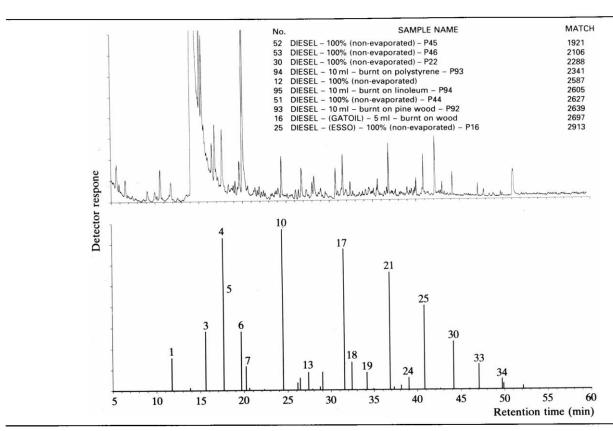


FIGURE 6 Total ion chromatogram and target compound chromatogram for a diesel sample after combustion on a mixture of wood, synthetic foam and assorted plastic materials. Insert shows database search results. The numbers refer to target compounds listed in Table 2.

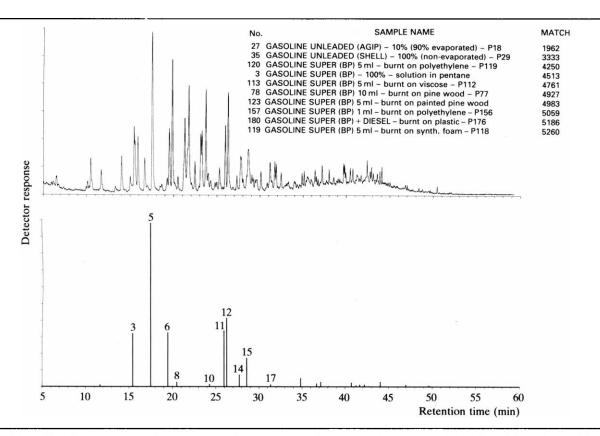


FIGURE 7 Total ion chromatogram and target compound chromatogram for a casework sample consisting of partially carbonized fire debris.

Insert shows database search results. The numbers refer to target compounds listed in Table 2.

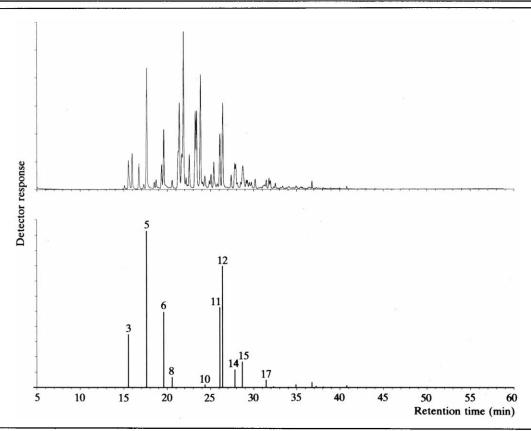


FIGURE 8 Total ion chromatogram and target compound chromatogram for a 90% evaporated petrol sample. The numbers refer to target compounds listed in Table 2.

results. Since the completion of our project, an article has been published by Holzer and co-workers [7] describing the design criteria for a GC-MS based expert system for arson analysis. The general approach described by these authors appears similar to our own, but their algorithm is much more complex and lengthy.

Acknowledgments

The authors would like to thank Raymond Keto, senior chemist at the National Laboratory Center, Bureau of Alcohol, Tobacco and Firearms (Rockville, Maryland, USA), for supplying additional information on his published TCC technique.

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